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Robert L. Fusaro

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Lubrication and Failure Mechanisms of Graphite Fluoride Films

Robert L. Fusaro
Lewis Research Center
Cleveland, Ohio

National Aeronautics
and Space Administration

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LUBRICATION AND FAILURE MECHANISMS OF GRAPHITE FLUORIDE FILMS

by Robert L. Fusaro

Lewis Research Center

SUMMARY

An optical microscope, equipped with a vertical illuminator and two polaroid filters (one rotatable), was used to visually study the sliding surfaces of 440C HT steel lubricated with rubbed films of graphite fluoride. In addition to visually observing the surfaces, the principles of light interference and birefringence of anisotropic crystals were used to study transfer, ordering, and the flow properties of graphite fluoride on the sliding surfaces. These observations were made at preset intervals throughout the wear life of the films by stopping the tests and removing the specimens from the apparatus.

Friction and wear results were compared to the visual observations for graphite fluoride films applied to three surface finishes - polished, sanded, and sandblasted. It was found that during the "run-in" of the films very smooth flat areas were created on the rider and on metallic asperities in the rubbed film wear track. The lubricating mechanism was then one of very thin films of graphite fluoride being sheared between these flat areas on the two opposing surfaces. The valleys on the roughened surface served three purposes: they tended to restrict the radial flow of the graphite fluoride out of the contact area, they served as a reservoir for supplying graphite fluoride to the contact area, and they served as a "dump" for metallic wear particles.

On "rough" surfaces the lubricating process consisted of the graphite fluoride being supplied from the valleys to the flat plateaus - in essence, flowing across the plateaus and being deposited in a following valley, etc. A certain amount of radial flow at the sides of the film wear track also took place; thus, as repeated passes were made over the film, the amount of lubricant available for lubrication was gradually depleted. When this happened, wear to the flat plateaus occurred so that the new lubricant deeper in the valleys could be used.

Failure was not abrupt; in general, friction and wear increased gradually as a function of repeated passes over the films. At some point, however, regions within the film wear track became depleted of graphite fluoride with the result that excessive metallic wear particles were produced. The metallic wear was fine and powdery, and it combined with graphite fluoride from other regions in the film that were not depleted to produce thick, powdery bands on both the rider and the disk wear track. Essentially, the formation of these bands marked the beginning of failure. Although lubrication was still achieved, the friction trace became rougher and rider wear increased at a faster rate.

INTRODUCTION

Graphite fluoride ($(CF_x)_n$) has been shown to have very good lubricating properties under various conditions and types of applications. References 1 and 2 show that rubbed films of graphite fluoride perform better than or equivalent to rubbed films of molybdenum disulfide (MoS_2) or graphite in tests conducted on a pin-on-disk machine. These rubbed film results were further improved by bonding graphite fluoride to the surface using a polyimide (ref. 3) or an organopolysiloxane (ref. 4) polymer. In reference 5, epoxy phenolic and silicate were used to bind graphite fluoride to surfaces which were then evaluated in a Falex machine.

Besides thin films, other areas where good results have been obtained with graphite fluoride are as additives in greases, mechanical carbons, and carbon fiber reinforced polytetrafluoroethylene (ref. 6); as an additive in graphite fiber reinforced polyimide (ref. 7); and as additives in films of graphite fluoride and metals which were simultaneously applied to a surface by using an electroplating bath (ref. 8). Although many good lubricating results have been reported with graphite fluoride, poor results have also been reported (refs. 9 and 10). The poor results were attributed to the abrasiveness (ref. 9) and low load carrying capacity (refs. 9 and 10) of graphite fluoride.

Most of the lubricating theories, the methods of evaluating, and the methods of applying and using solid lubricants have been based on studies of MoS_2. To date, very little research has been conducted on the fundamental lubricating mechanisms of graphite fluoride and how they compare to those of MoS_2. It might be that what is good for MoS_2 is not good for graphite fluoride.

Thus, the purpose of this investigation was to obtain a more fundamental understanding of how graphite fluoride lubricates and how failure occurs. The scope of this work included studying the effect of surface finish (onto which rubbed films of graphite fluoride were applied) on friction coefficient, wear life, and rider wear, and then comparing these results with observations made of the sliding surfaces by optical microscopy. This was accomplished by stopping the tests at preset intervals, removing the specimens from the apparatus, and visually studying the surfaces with the microscope. The microscope was equipped with a vertical illuminator and two polaroid filters (one rotatable). Thus, the principles of light interference and birefringence of anisotropic crystals were used to study transfer, ordering, and the flow properties of the graphite fluoride films.

The experimental conditions used were a pin-on-disk test apparatus, a controlled atmosphere of moist air (10 000 ppm H_2O), a test temperature of 25° C, a sliding speed of 2.6 meters per second (1000 rpm), and a load of 1 kilogram.

MATERIALS

The graphite fluoride used in this investigation had a fluorine to carbon ratio as obtained from the manufacturer of 0.85 to 1.00. A previous investigation (ref. 11) showed that higher fluorine content graphite fluoride compounds do not give appreciably better lubrication results and are considerably more difficult to make and more expensive to purchase. The average particle size of the graphite fluoride used was 10 micrometers, but the size ranged from less than 1 micrometer up to 30 micrometers; however, the larger particles appeared to be conglomerations of smaller particles.

The riders and disks were made of 440C HT steel with a Rockwell hardness of C-60. The disks were lapped and polished to a surface roughness of 0.09±0.02 micrometer CLA (centerline average). The graphite fluoride was applied to the polished disk and to disks that were roughened (by sanding in random directions with number 150 grit wet sand paper) to 0.30±0.05 micrometer (CLA) and to disks that were roughened (by sandblasting) to 1.2±0.2 micrometer (CLA).

The graphite fluoride was applied to the roughened or polished surfaces by mechanically rubbing it over the surface at constant load (see PROCEDURE section). The thickness of the films obtained, as determined by surface profile and optical microscope studies, was found to be of the order of 1 micrometer above the highest feature on the metallic surface.

APPARATUS

A pin-on-disk sliding friction apparatus was used in this study. This apparatus has been described in previous reports (refs. 1 to 4). Basically, the friction specimens (fig. 1) consisted of a flat disk (6.3-cm diameter) in sliding contact with a stationary hemispherically tipped rider (0.476-cm radius). The rider slid on a 5-centimeter-diameter track on the disk, which gave the rider a linear sliding speed of 2.6 meters per second at a disk rotation of 1000 rpm.

The apparatus used to apply the solid lubricant powder to the disks is shown in figure 2. The disk was attached to the vertical shaft of a small electric motor by a cup-shaped holder. Two vertical rods were used to restrain a floating metal plate to which was attached the solid lubricant applicator. In these experiments the backs of polishing cloths were used as applicators. The load was applied by placing weights on top of the metal plate.

The burnishing apparatus was designed to fit under the bell jar of a vacuum system; thus, the atmosphere in which burnishing took place could be controlled by pumping a vacuum and then backfilling with the desired atmosphere. Previous results (ref. 11) have shown that films applied in moist air (10 000 ppm H_2O) give better friction and wear

results than films applied in dry air (<20 ppm H_2O); thus, the films used in these experiments were applied in moist air.

PROCEDURE

Surface Cleaning

The cleaning procedure after the desired surface finish was given to the disks was as follows:

(1) Scrub the surface under running tap water with a brush to remove abrasive particles.

(2) Clean the surface with pure ethyl alcohol.

(3) Rub the surface with a water paste of levigated alumina. Clean until water wets the surface readily.

(4) Rinse the surface under running tap water to remove levigated alumina (use a brush to facilitate removal).

(5) Rinse the surface in distilled water.

(6) Dry the surface using dry compressed air. (Surface not dried quickly has a tendency to oxidize).

The riders were also cleaned by this procedure, but, since only polished riders were used, step (1) was not necessary.

Film Application

The procedure for applying the rubbed films was as follows:

(1) Apply a small amount of graphite fluoride powder to the cleaned disk surface and spread it evenly over the surface with the back of a polishing cloth.

(2) Apply about 0.5 gram of graphite fluoride to the contact zone of the applicator (back of a polishing cloth attached to the floating metal plate) and distribute it evenly.

(3) Assemble the apparatus as shown in figure 2 and apply two 1-kilogram weights for the applied load.

(4) Evacuate the bell jar and backfill it with an atmosphere of moist air (10 000 ppm H_2O). Continue to purge the bell jar with moist air until the disk is removed from the apparatus.

(5) Set the disk into rotation by gradually increasing the speed to 15 rpm and rub for 1 hour.

(6) Remove the disk from the apparatus and blow the loose graphite fluoride debris from the surface using dry compressed air.

Friction and Wear Tests

A rider and disk (with applied graphite fluoride film) were inserted into the apparatus and the test chamber was sealed. Moist air (10 000 ppm H_2O) was purged through the chamber for 15 minutes before starting the tests; this purge was continued throughout the tests. Moist air was used as a controlled atmosphere, since this is typical of average atmospheric conditions (approximately 50 percent relative humidity). The flow rate was 1500 cubic centimeters per minute, and the volume of the chamber was 2000 cubic centimeters. After purging for 15 minutes, the disk was set into rotation at 1000 rpm and a 1-kilogram load was gradually applied. The test temperature was 25° C.

Each test was stopped after 1 kilocycle (1 minute) of sliding. After the rider and disk were removed from the friction apparatus the contact areas were examined by optical microscopy and photographed. Surface profiles of the disk wear tracks were also taken. The rider and disk were then placed back into the apparatus and the previous test procedure was repeated. The rider was not removed from the holder, and locating pins in the apparatus insured that it was returned to its original position. The same was true for the disk.

Each test was stopped and the previous procedure repeated after sliding times of 1, 5, 15, 30, 60, 100, 200, 400, 800, and 1200 minutes, or when failure occurred. The failure criterion in this study was a friction coefficient of 0.25. Rider wear was determined by measuring the diameter of the wear scar on the hemispherically tipped rider and then calculating the volume of material worn away.

Analysis of Sliding Surfaces

Optical microscopy techniques were used to study the lubricating films, the transfer films, and the wear particles in this investigation. The surfaces were viewed at magnifications to 1100. At these high magnifications, the depth of field was very small (of the order of 1 μm); thus, this aspect was used to advantage in measuring the heights of various features on the sliding surfaces such as film thickness, wear track depth, etc.

The thin films of graphite fluoride were transparent. And since illumination and observation of the surfaces were normal to the surfaces, interference fringes could be seen in the graphite fluoride films both on the disk wear track and on the rider. Interference fringes indicated that graphite fluoride was present and that the films were smooth and continuous.

The microscope was also equipped with two polaroid filters (one which could be rotated); thus, the sliding surfaces could be examined between cross polaroid filters.

Since graphite fluoride was birefringent and the metallic wear debris was not, this technique was used to distinguish graphite fluoride particles from metallic wear debris.

RESULTS AND DISCUSSION

Friction, Wear, and Wear Life

The graphite fluoride powder was applied to 440C HT steel disks with three different surface finishes: lapped and polished to a surface roughness of 0.09±0.02 micrometer (CLA), sanded to a surface roughness of 0.30±0.05 micrometer (CLA), and sandblasted to a surface roughness of 1.2±0.2 micrometer (CLA). Figure 3 gives (a) photomicrographs and (b) surface profiles of the three different surfaces to which the graphite fluoride was applied. The figure illustrates the large variations in the roughness of the surfaces. The techniques used to roughen the surfaces and apply the graphite fluoride are given in the PROCEDURE section.

Figure 4 gives friction traces for 440C HT riders sliding on the graphite fluoride films applied to the three different surface finishes. The tests were terminated when the friction coefficient exceeded 0.25. As the figure illustrates, the surface finish of the substrate, to which the graphite fluoride was applied, was very critical in determining the life of the films. Wear life increased with increasing surface roughness; the polished surface gave a wear life of 42 kilocycles, the sanded surface gave a wear life of 380 kilocycles, and the sandblasted surface gave a wear life of 1250 kilocycles. Even though the sandblasted surface of CLA roughness of 1.2±0.2 micrometers gave the longest wear life, this may not be the optimum surface finish. The main objective of this report was to investigate the mechanisms of graphite fluoride lubrication, and the three different surface finishes used were randomly chosen to show the lubricating mechanisms.

It was found that the smoother the surface the lower the initial friction coefficient for the three surface finishes evaluated. The values obtained were, respectively, 0.04, 0.05, and 0.07. The difference was most likely due to a "run-in" effect caused by the rougher surfaces having a greater number of metallic asperities protruding through the rubbed film. During the initial phases of sliding these asperities were either plastically deformed or worn away, thus contributing to the initial differences in friction.

After run-in, the differences in friction coefficient (also wear life of the films and wear to the riders) were influenced by both the dynamics of the sliding interface and the texture of the surface to which the graphite fluoride was applied. The flow (or shear) properties of graphite fluoride were found to be excellent, and with the polished surface the graphite fluoride tended to flow from the contact zone much more readily than with the rougher surfaces. Thus, a rough surface tended to restrict the radial flow of graphite fluoride from the contact zone. The rough surfaces also served as a reservoir for

graphite fluoride and as a "dump" for fine metallic wear debris. These aspects of the lubricating process will be discussed in more detail in following sections.

At preset intervals throughout the experiments the tests were stopped to remove the rider and disks specimens from the apparatus to examine the rubbing surfaces. The diameter on the circular scar on the rider was measured and from this was calculated the volume of material worn from the rider. Figure 5 gives rider wear volume as a function of sliding duration (in kilocycles) for the riders which slid on graphite fluoride films applied to the three different surface finishes. The rider wear rate was calculated for each interval the tests were stopped, and table I gives those data in terms of wear volume per unit distance of sliding.

As found for the friction coefficient, the rougher the surface the higher the initial rider wear. Again, this was most likely due to the run-in process. The rougher the surface, the larger the number of metallic asperities that stuck through the film - thus, the higher the rider wear. As figure 5 and table I illustrate, this run-in process seemed to last longer on rougher surfaces. It took more than 15 kilocycles of sliding before wear to the rider which slid on the sandblasted surface leveled off; the wear to the rider which slid on the sanded surface leveled off after about 5 kilocycles of sliding. With the polished surface it was hard to specify if there was a run-in process taking place at all, since wear after 1 kilocycle of sliding was constantly increasing. Thus, if there were a run-in process, it must have been less than 1 kilocycle.

In general, for the rough surfaces (sandblasted and sanded), the rider wear process consisted of three regimes. The first was an interval of gradually decreasing wear rates, the second was an interval or relatively constant wear rates, and the third was an interval of gradually increasing wear rates. These regimes are discussed later in terms of the lubricating mechanisms taking place.

The wear of the disk surfaces, to which the graphite fluoride was applied, was also studied. Figure 6 gives surface profiles of these wear tracks which were taken after 1, 15, 60, and 200 kilocycles of sliding. A profile is also shown of the wear track on each surface after failure has occurred. Since the film on the polished surface failed after 42 kilocycles, three surface profiles are shown.

The vertical magnification of the surface profiles is about 75 times the magnification of the horizontal; thus, a distorted view of the true surface was obtained. The surface profiles indicate that the sliding surface of the wear track tended to be very smooth, at least until failure occurred. At failure the wear track was found to be somewhat rough, due to the buildup of powdery metallic debris. On the polished surface and on the sanded surface, very little wear was observed to occur to the metallic disk. From the surface profiles, metallic wear was not distinguishable from film wear. On the sandblasted surface, however, there is a very prominent depth to the wear track. Surface profile studies and optical microscope studies of the films (applied to all three surface finishes)

indicated that the thicknesses of the films were of the order of 1 micrometer above the highest feature of the metallic surface. Thus, for the sandblasted surface, the depth of the wear track at failure was approximately 6 micrometers below the highest asperities on the disk sandblasted surface.

Lubrication of Sandblasted Surfaces

The lubricating mechanisms of the graphite fluoride films were studied by optical microscopy. As mentioned previously, the tests were stopped at various sliding intervals and wear measurements were taken. During this time the surfaces were also studied by optical microscopy and with crossed polaroid filters.

Figure 7 shows the wear track on the graphite fluoride film applied to the sandblasted disk after (a) 1, (b) 5, (c) 60, and (d) 800 kilocycles of sliding. These low magnification photomicrographs illustrate that the film in the wear track does not uniformly cover the metal surface. The bright spots on the film are sandblasted metal asperities that stick through the film. As figure 7 illustrates, the number of these asperities increased as repeated passes were made over the wear track, and the size of each asperity also increased. Comparing figure 7 with figure 4 shows that even though the amount of metallic contact was increasing the friction coefficient was relatively uneffected. The rider wear rate, however, reached a minimum between 100 and 200 kilocycles of sliding and then increased gradually with sliding distance (table I).

In an attempt to understand the mechanism by which graphite fluoride was providing lubrication, the contacting surfaces were observed at higher magnifications up to a maximum of 1100. Figure 8 shows high magnification photomicrographs of the central portion of the graphite fluoride film wear tracks after (a) 1, (b) 5, (c) 100, and (d) 1200 kilocycles of sliding. The figure illustrates that the bright areas of figure 7 are in reality flat metallic plateaus which are covered with extremely thin films of graphite fluoride.

The graphite fluoride can be discerned on the metallic plateaus by color changes and interference fringes. In some instances the graphite fluoride flows across the asperities in layers thick enough to show a distinct edge to the layer (fig. 8(d)); in most cases, however, the films gradually blend into the surfaces, being sheared thinner and thinner (fig. 8(b)).

Photomicrographs of the rider scars which correspond to the film wear tracks of figure 7 are shown in figure 9. One noticeable feature of the rider scars is the absence of a heavy transfer film. The scars are extremely smooth and the transfer that is present is extremely thin.

Figure 10 shows high magnification photomicrographs of typical transfer films in various regions of the rider scar after various sliding intervals. Where the film is

thick enough or continuous enough, interference fringes can be seen in the transfer. An illustration of this is shown in figure 10(a), where the entrance area is seen. As the graphite fluoride moves into the contact zone on the rider (flat area), it is sheared thinner and thinner until it is too thin or not continuous enough for interference fringes to be seen. The graphite fluoride flows through the contact zone of the rider and, as figure 10(b) illustrates, tends to coalesce into thicker films when leaving the contact area.

During the initial stages of sliding when contact stresses are high (small rider scars) and an abundant supply of lubricant is available, the transfer film within the contact area is very thin and fairly uniform. No interference fringes are seen and the transfer appears milky colored when viewed under the microscope at high magnifications (figs. 10(b) and (c)). Since no fringes are seen, this indicates the thickness of the films are less than the wavelength of light (0.4 μm). At later stages of sliding, the contact stresses are reduced (due to wear of the rider) and thicker films are observed in the middle of the rider scar. Figure 10(d) shows a transfer to the middle of the scar after 800 kilocycles of sliding. Some interference fringes can be seen, indicating heavier transfer, but the film is somewhat spotty because the graphite fluoride is being depleted from the contact zone.

How the lubricating mechanism of graphite fluoride films applied to sandblasted surfaces appears to be is now described. When the rider and graphite fluoride film are initially brought into sliding contact, very high contact stresses develop on the surface asperities of the sandblasted metal. To accommodate this stress, a high wear condition exists and the asperities either plastically deform or wear until the contact area is such that it will support the load. After this run-in the lubrication mechanism is one of very thin films of graphite fluoride being sheared across these flat plateaus that were produced during the run-in. Thus, the lubrication process is of a dynamic nature, the graphite fluoride is supplied to the plateaus from the valleys which are filled with graphite fluoride powder, and very thin films of graphite fluoride essentially flow across the flat sandblasted plateaus and are deposited in the following valley.

An analogy might be made with the rider, which in effect is also a flat plateau but much larger. The graphite fluoride, which is supplied from the buildup of lubricant in the entrance region, flows across the flat rider scar and is deposited in the exit region (figs. 9 and 10). Flow also takes place radially at the sides of the film wear track; thus, as repeated passes are made over the film, the amount of lubricant available for lubrication is gradually depleted. As this happens, wear to the sandblasted flat plateaus must occur to expose new lubricant deeper in the valleys. Thus, as seen in figures 7 and 8, the amount of metal on which the film shears gets progressively larger. As the friction trace for the rider sliding on the sandblasted surface illustrates, this does not markedly affect the friction coefficient since it increases very slowly with repeated passes of sliding.

In essence, the graphite fluoride is supplied to the flat plateaus from two sources, either the valleys on the sandblasted disk or the buildup of lubricant in the entrance area of the rider scar. The graphite fluoride particles get trapped between two flat surfaces and shear and thus provide lubrication. This process is very similar to what Sliney (ref. 12) observed when he viewed various lubricants and abrasives sliding between a glass disk and a metal ball.

Lubrication of Sanded Surfaces

In an attempt to better understand the role of the surface finish (to which the graphite fluoride films were applied) in the lubrication mechanism, graphite fluoride films were applied to 440C steel surfaces that were roughened by sanding. The same experimental conditions that were used when evaluating graphite fluoride films on sandblasted surfaces were used with these films.

Figure 11 shows the wear track on the graphite fluoride film that was applied to the sanded disk after (a) 1, (b) 5, (c) 60, and (d) 200 kilocycles of sliding. The amount of metal observed in the wear track on the sanded disk increased as the number of sliding cycles increased; these findings are similar to those obtained when sandblasted surfaces were used. It is also seen that the graphite fluoride tended to flow radially out of the contact area.

Figure 12 shows high magnification photomicrographs of the graphite fluoride wear tracks for the same sliding intervals. The flowing nature of the film is apparent, and after only one kilocycle of sliding (fig. 12(a)) the graphite fluoride has been sheared into a very thin film which shows a multitude of colorful interference fringes. The scratches in the metallic surface (caused by sanding) are filled with graphite fluoride, and this material is sheared extremely thin over the plateau areas between the scratches. In some instances, a chunk of the compressed graphite fluoride material will be disengaged from the scratch areas (fig. 12(b)), but for the most part the graphite fluoride is gradually fed from the reservoirs to the flat plateaus.

As sliding continued, the amount of graphite fluoride was gradually depleted, and wear to the flat metal plateaus occurred and exposed more lubricant deeper in the scratches. Since the sanded surfaces did not have the reservoir capacity of the sandblasted surfaces, the life of the film was much shorter (fig. 4). Even though continual wear of the metal surfaces did occur, the graphite fluoride prevented galling wear on the surfaces. This can be observed in figure 11(d) where the sanded surface is seen to become smoother and smoother as the graphite fluoride is being depleted.

As far as the rider surfaces were concerned, the formation of transfer films and the wear to the rider took place almost identically to those riders which slid on films applied to sandblasted surfaces. The only differences were that the sanded surface was less

abrasive during run-in and wear to the rider was lower in the early stages of sliding (table I).

Lubrication of Polished Surfaces

As a further check on the effect of surface finish on the lubrication process, graphite fluoride films were applied to 440C disks that were lapped and polished to a surface roughness of 0.09±0.02 micrometer and that essentially contained no lubricant reservoirs. Figure 13 shows photomicrographs of rider and disk wear tracks after 1 kilocycle of sliding. Heavier transfer was seen on the rider, and the film on the disk wear track was less continuous than for the films applied to the rougher surfaces.

Figure 14 shows high magnification photomicrographs of the inner edge of the disk wear track after (a) 1 and (b) 5 kilocycles of sliding. The lubrication mechanism on the smooth surface was one of individual graphite fluoride particles getting sheared into very thin, irregular shaped platelets. These platelets separate the two metallic surfaces, and they flow as relative motion takes place. Unfortunately, when the surface was smooth, there was nothing to constrict their lateral movement so they flowed out of the contact area fairly quickly. Figure 14 shows the depletion of the film after 1 and 5 kilocycles of sliding and the coalescing of the platelets at the edge of the disk wear track.

Failure Mechanisms

As a general rule, failure in these experiments was a gradual process. After the run-in and an interval with a relatively constant friction coefficient, the friction coefficient gradually increased with sliding distance. The same was true for rider wear. At some point, however, depending on surface roughness, the friction trace (fig. 4) got much rougher and there was an increase in rider wear rate. This occurred for the polished surface after about 15 kilocycles of sliding, for the sanded surface after about 200 kilocycles of sliding, and for the sandblasted surface after about 1100 kilocycles of sliding (fig. 4 and table I).

What happened on the surfaces when this occurred was quite interesting. As discussed previously, a certain amount of metallic wear occurred to both the rider and the flat metallic asperity plateaus on the disk. The metallic wear particles were found to be extremely fine and often were not easily distinguishable from the graphite fluoride particles. To ascertain what particles were graphite fluoride and which were metal, the surfaces were viewed between crossed polaroid filters. Since graphite fluoride particles were birefringent and the metal particles were not, only the graphite fluoride

particles were visible. Thus, by crossing and uncrossing the filters, metallic particles could be distinguished from graphite fluoride particles.

Even though metallic particles were being constantly produced, they did not seem to be of any great consequence to good lubrication as long as an ample supply of graphite fluoride was available. The metal particles tended to find there way out of the contact area at the sides of the wear track or they were deposited in the valleys between the plateaus on the rough surfaces.

At some point during the test, usually marked by rougher friction, regions on the disk wear track became depleted of graphite fluoride. Figure 8(d) shows a valley nearly depleted of graphite fluoride. When too many of these valleys became depleted, excessive metallic wear particles were produced. These wear particles mixed with graphite fluoride particles still present in other regions, and a powdery mixture of the two constituents resulted. The powdery mixture extended in a band (or bands) which ran around the complete circumference of the disk wear track. A band corresponding to this was also found on the rider. Photomicrographs of these bands are shown in figure 15 for the (a) sandblasted, (b) sanded, and (c) polished surfaces which were run to failure. It is interesting to note that the surfaces underneath this powdery buildup were still very smooth and no galling wear had taken place.

Figure 16 shows high magnification photomicrographs of the powdery films found on the sanded surface after 300 and 380 kilocycles of sliding. The figure illustrates the progression of failure. Since at 300 kilocycles the powdery mixture consisted mostly of graphite fluoride, fairly good flow properties are seen on both the rider (fig. 16(a)) and the disk wear track (fig. 16(b)). After 380 kilocycles of sliding, however, the flow properties of the film on the rider (fig. 16(c)) and on the disk wear track (fig. 16(d)) are not nearly as good. The transfer looks darker and more powdery. When examined under crossed polaroid filters, hardly any birefringent particles can be seen.

Thus, the failure mechanism for the graphite fluoride films appears to be the depletion of the graphite fluoride from the contact zone by flowing radially outward. This was followed by the excessive production of metallic debris, which combined with the graphite fluoride to form a highly adherent powdery mixture. This mixture, however, was still able to provide lubrication; but, as the mixture was depleted of graphite fluoride, friction and wear also increased.

Besides radial flow, a second mechanism for depletion of graphite fluoride has been found by Atkinson and Waghorne (ref. 13). They studied the sliding surfaces of graphite fluoride films applied to steel using X-ray photoelectron spectroscopy and deduced that failure was caused by the chemical degradation of graphite fluoride and the destruction of the C-F lattice structure. Most likely this mechanism also contributed to the depletion of the film in this study - especially on the sandblasted surface where radial flow was restricted by surface roughness.

SUMMARY OF RESULTS

Friction, wear, and optical microscopy studies of graphite fluoride rubbed films applied to surfaces with different surface finishes indicated the following:

1. The lubrication mechanism consisted of the shear of graphite fluoride particles between flat areas on the rider and on metallic plateaus in the film wear track area. The flat areas were created during run-in; the sheared graphite fluoride particles tended to "flow" between these flat surfaces having an entrance region and an exit region. Thus, the lubricating process was a dynamic one.

2. Wear life was inhanced by restricting the radial flow of the graphite fluoride from the contact area by increasing the surface roughness. Valleys in the roughened surfaces also served as reservoirs for graphite fluoride and as dumps for metallic debris; thus, wear life increased with increased reservoir capacity.

3. Failure (high friction and wear) was caused by the depletion of the graphite fluoride from the contact area and by the excess formation of fine metallic debris, which combined with the remaining graphite fluoride to form heavy, powdery transfer bands on both sliding surfaces. No galling wear to the surfaces was observed.

Lewis Research Center,
National Aeronautics and Space Administration,
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505-04.

REFERENCES

1. Fusaro, Robert L.; and Sliney, Harold E.: Preliminary Investigation of Graphite Fluoride $(CF_x)_n$ as a Solid Lubricant. NASA TN D-5097, 1969.

2. Fusaro, Robert L.; and Sliney, Harold E.: Graphite Fluoride $(CF_x)_n$ - A New Solid Lubricant. ASLE Trans., vol. 13, no. 1, Jan. 1970, pp. 56-65.

3. Fusaro, Robert L.; and Sliney, Harold E.: Graphite Fluoride as a Solid Lubricant in a Polyimide Binder. NASA TN D-6714, 1972.

4. Fusaro, Robert L.: Organopolysiloxane-Bonded Graphite Fluoride as a Solid Lubricant. NASA TN D-8033, 1975.

5. Gisser, H.; Petronio, M.; and Shapiro, A.: Graphite Fluoride as a Solid Lubricant. Lubr. Eng., vol. 28, no. 5, May 1972, pp. 161-164.

6. Ishikawa, T.; Teranishi, H.; and Morishita, M.: Carbon Fiber, and Composite Materials of Carbon Fibre and Polytetrafluoroethylene. Carbon Fibers, Their Composites and Applications, Plastic Inst. (London), 1971, Paper no. 35.

7. Fusaro, Robert L.; and Sliney, Harold E.: Friction and Wear Behavior of Graphite Fiber Reinforced Polyimide Composites. NASA TM X-73565, 1977.

8. Tsuya, Yoko; et al.: Codeposited Composite Metal-Graphite Fluoride Platings. ASLE Trans., vol. 17, no. 3, 1974, pp. 229-235.

9. Mecklenburg, K. R.: The Effect of Wear on the Compressive Stress in the Sphere-on-Plane Configuration. ASLE Trans., vol. 17, no. 2, 1974, pp. 149-157.

10. McConnell, B. D.; Snyder, C. E.; and Strang, J. R.: Analytical Evaluation of Graphite Fluoride and Its Lubrication Performance Under Heavy Loads. Lubr. Eng., vol. 33, no. 4, Apr. 1977, pp. 184-190.

11. Fusaro, Robert L.: Graphite Fluoride Lubrication: The Effect of Fluorine Content, Atmosphere, and Burnishing Technique. ASLE Trans., vol. 20, no. 1, Jan. 1977, pp. 15-24.

12. Sliney, Harold E.: Dynamics of Solid Lubrication as Observed by Optical Microscopy. NASA TM X-71880, 1976.

13. Atkinson, I. B.; and Waghorne, R. M.: The Tribo-Chemistry of Graphite Fluoride Studied Using X-Ray Photoelectron Spectroscopy. Wear, vol. 37, 1976, pp. 123-128.

TABLE I. - EFFECT OF SURFACE FINISH ON RIDER WEAR RATE FOR RIDERS WHICH SLID ON RUBBED GRAPHITE FLUORIDE FILMS APPLIED TO 440C HT STEEL DISKS WITH DIFFERENT SURFACE FINISHES

[Experimental conditions: load, 1 kg; speed, 2.6 m/sec (1000 rpm); atmosphere, moist air (10 000 ppm H_2O), temperature, 25° C.]

Sliding interval, kilocycles	Rider wear rate, $\mu m^3/mm$		
	Sandblasted surface	Sanded surface	Polished surface
	Surface finish, μm		
	1.2±0.2	0.30±0.05	0.09±0.02
0 to 1	3.3	1.6	0.59
1 to 5	2.0	.43	.45
5 to 15	1.2	.03	.53
15 to 42	----	----	1.4
15 to 60	.13	.04	Failure
60 to 100	.12	.11	↓
100 to 200	.06	.12	↓
200 to 308	----	.45	↓
308 to 381	----	2.2	↓
200 to 400	.20	Failure	↓
400 to 800	.52	↓	↓
800 to 1200	.95	↓	↓
1200 to 1250	2.9	↓	↓

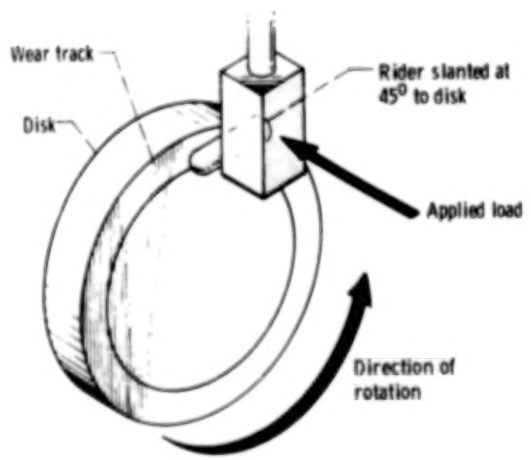


Figure 1. - Schematic diagram of friction specimens.

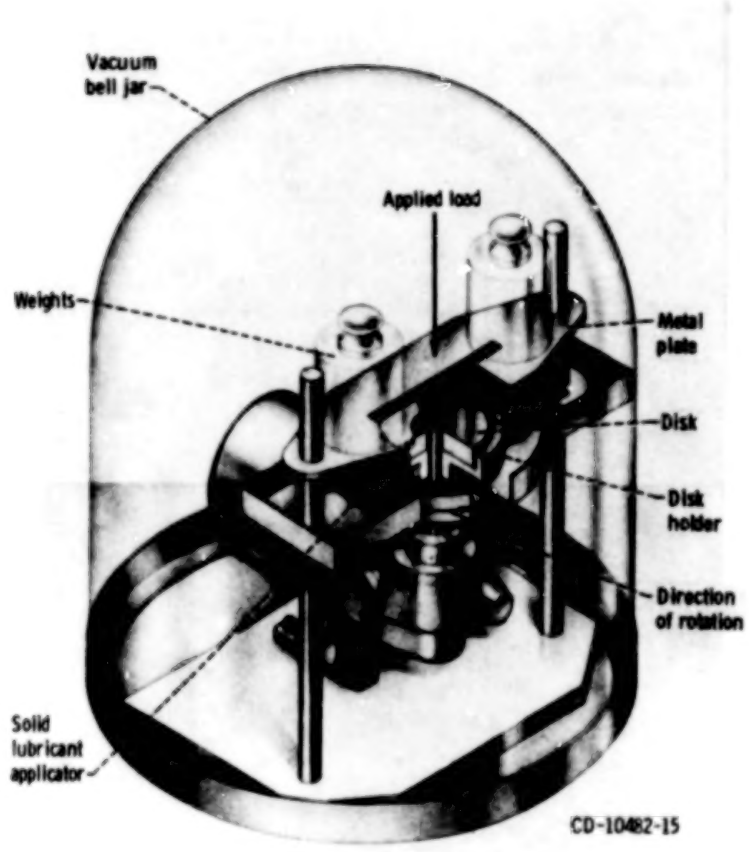
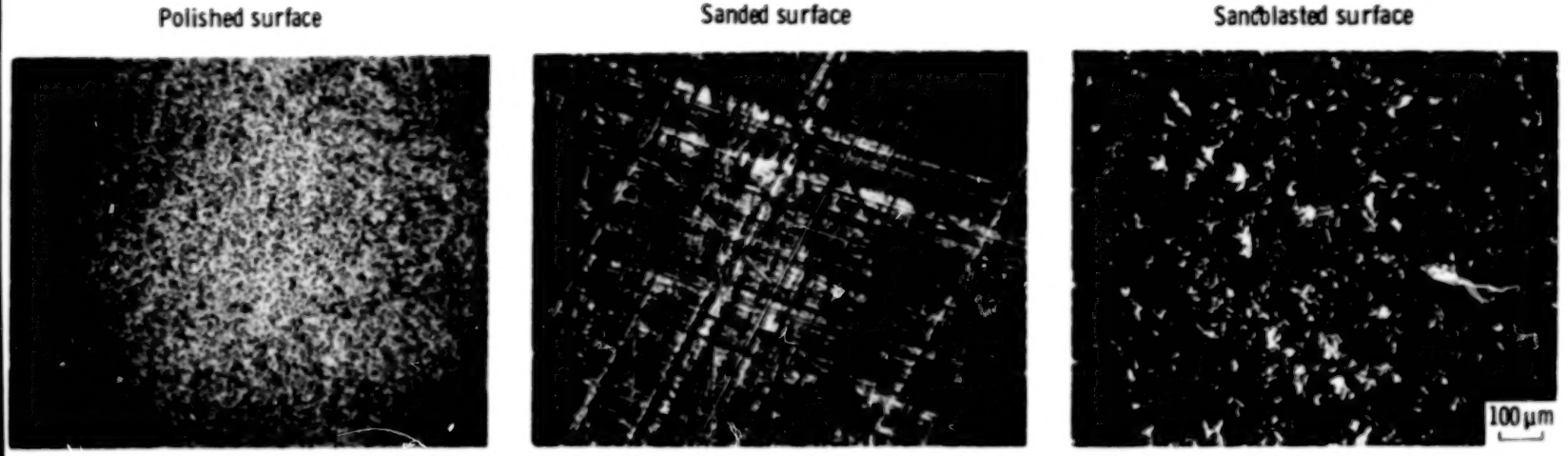
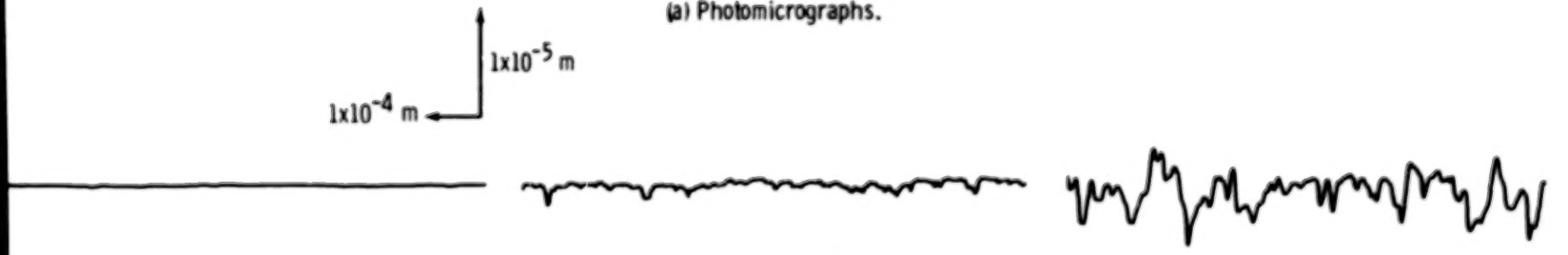


Figure 2. - Apparatus used for applying solid lubricant films.



(a) Photomicrographs.



(b) Surface profiles.

Figure 3. - Photomicrographs and surface profiles of three surfaces to which graphite fluoride was applied.

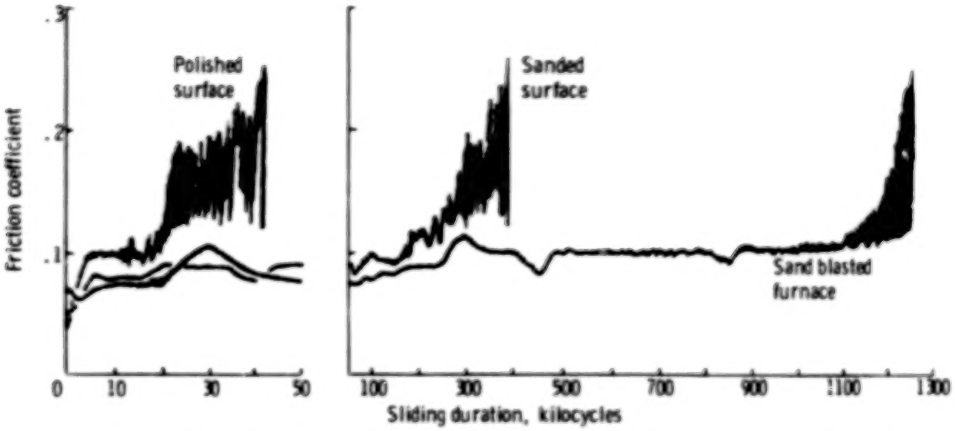


Figure 4. - Friction traces for graphite fluoride films rubbed onto disks with different surface finishes. Experimental conditions: load, 1 kilogram; speed, 2.6 meters per second; test temperature, 25° C; test atmosphere, moist air (10 000 ppm H_2O).

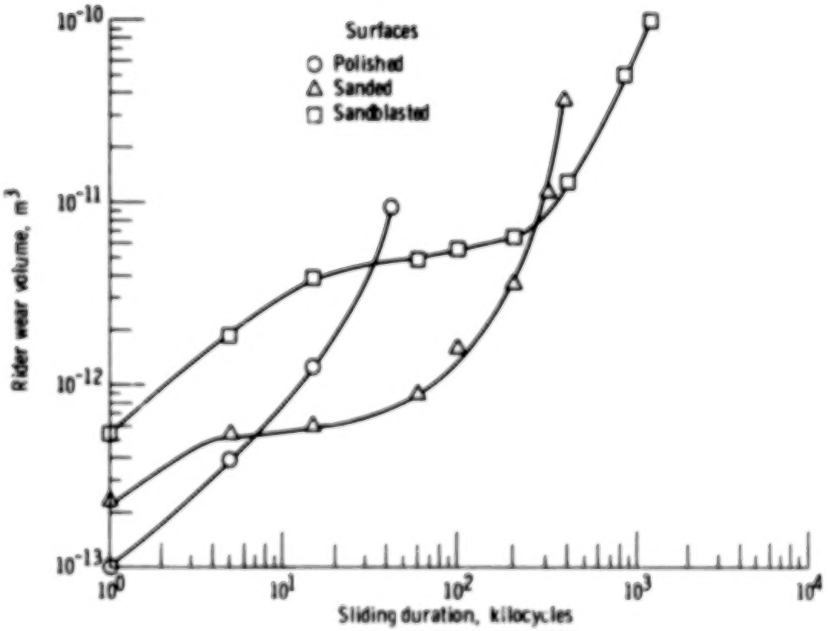


Figure 5. - Wear of riders which slid on graphite fluoride films rubbed onto disks with different surface finishes. Experimental conditions: load, 1 kilogram; speed, 2.6 meters per second (1000 rpm); test temperature, 25° C; test atmosphere, moist air (10 000 ppm H_2O).

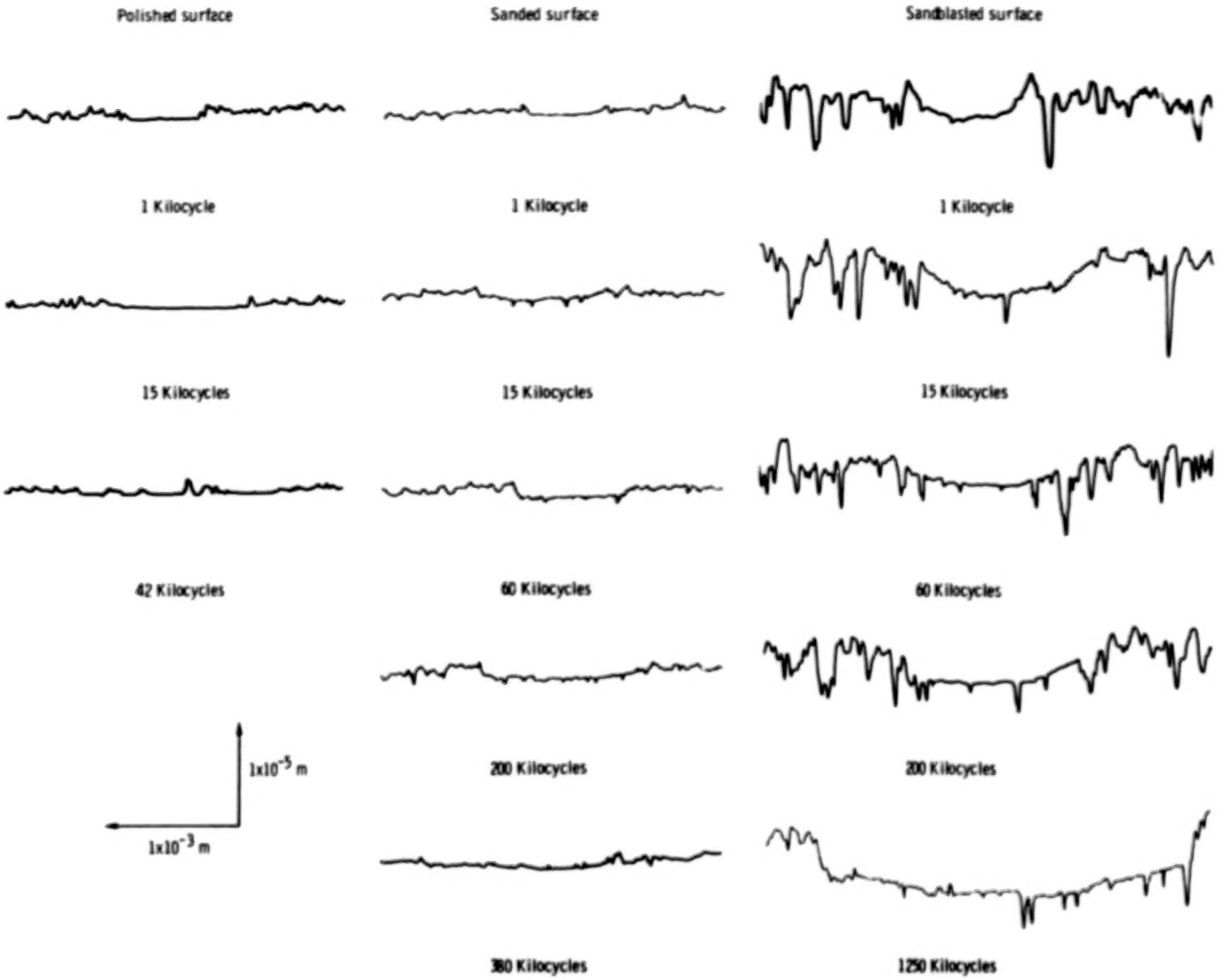
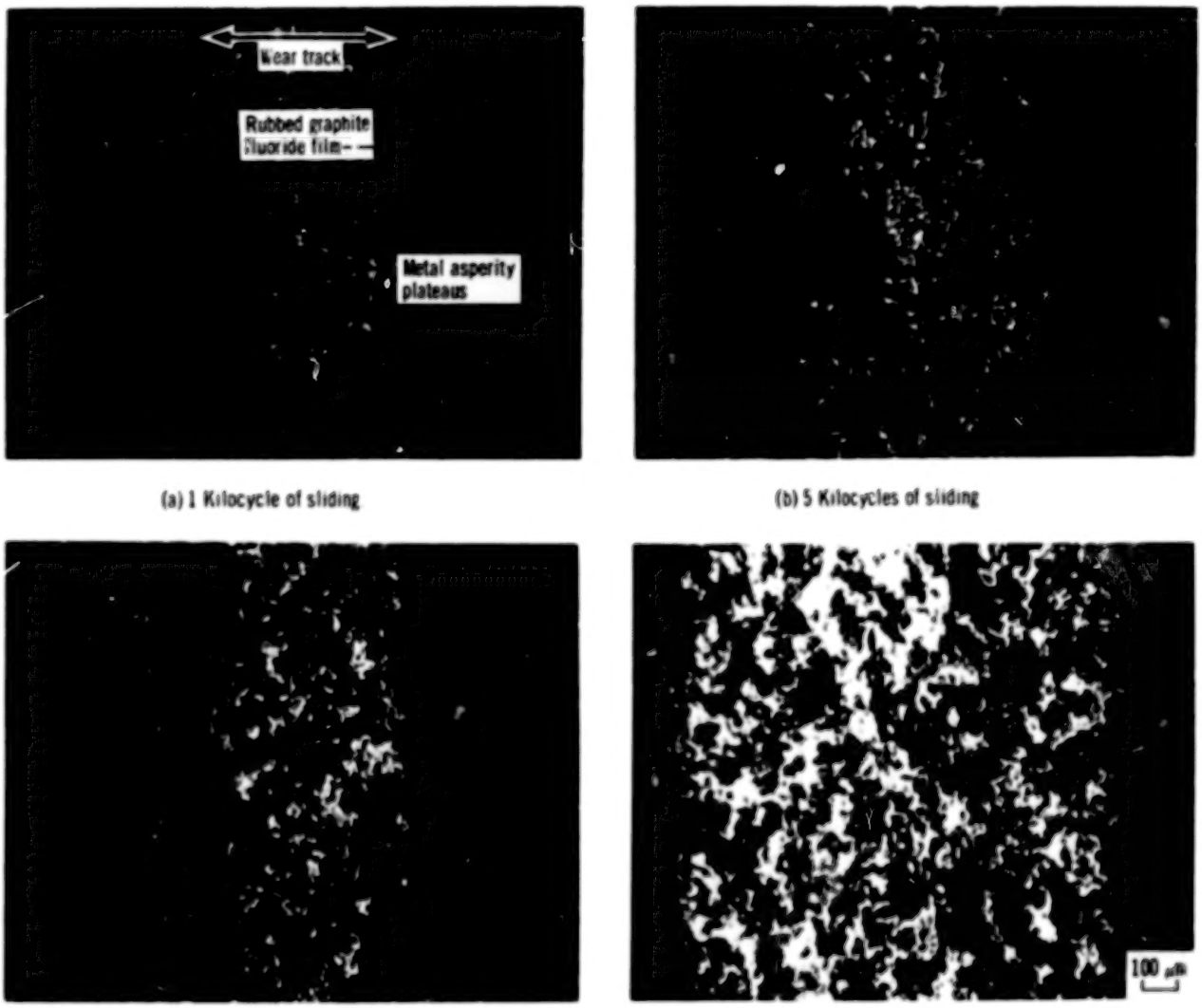


Figure 6. - Surface profiles of wear tracks on rubbed graphite fluoride films (taken after various sliding intervals) applied to 440C HT steel disks with three surface finishes. Experimental conditions: test temperature, 25° C; load, 1 kilogram; sliding speed, 2.6 meters per second (1000 rpm); atmosphere, moist air (10 000 ppm H_2O).



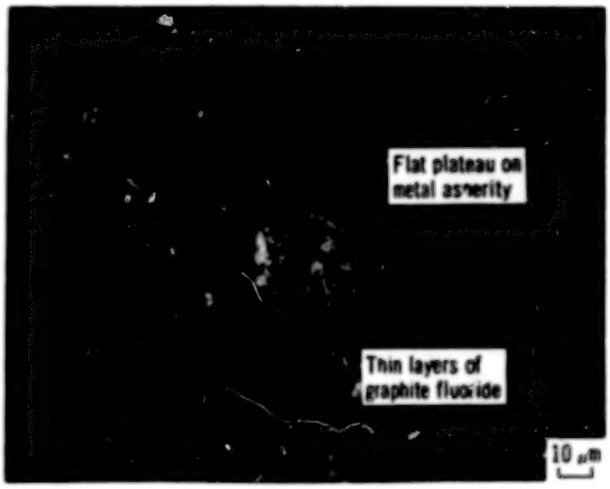
(a) 1 Kilocycle of sliding

(b) 5 Kilocycles of sliding

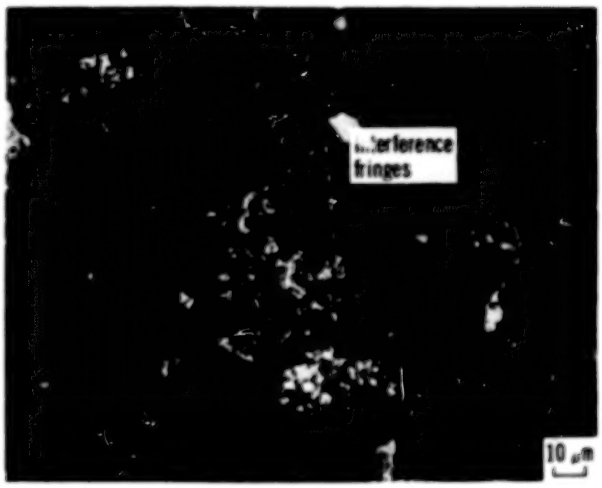
(c) 60 Kilocycles of sliding.

(d) 800 Kilocycles of sliding.

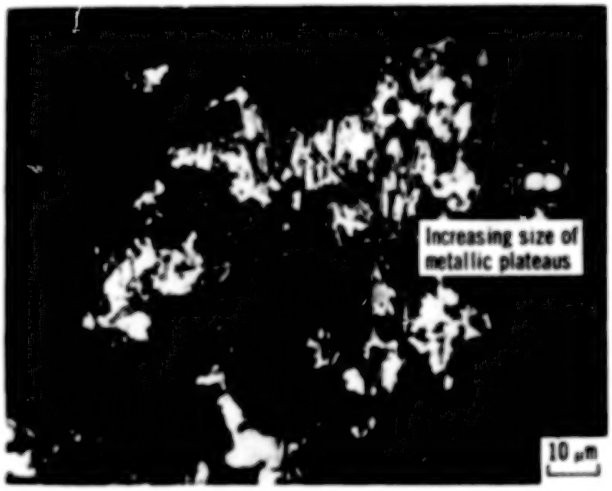
Figure 7. - Photomicrographs taken after various distances of sliding for graphite fluoride films applied to sandblasted 440C steel surfaces. Experimental conditions: load, 1 kilogram; speed, 2.6 meters per second; test temperature, 25° C; test atmosphere, moist air (10 000 ppm H_2O).



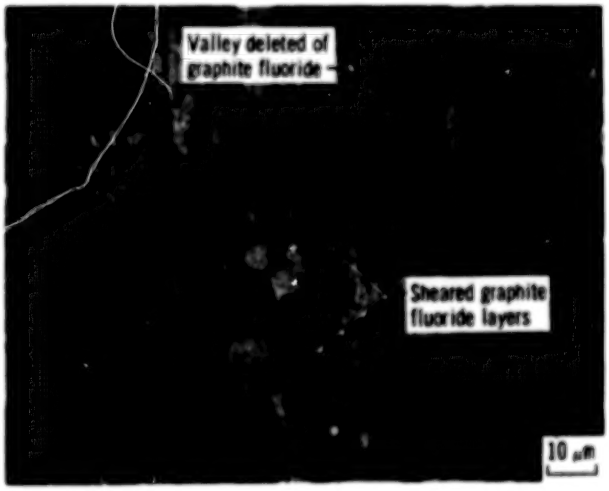
(a) 1 Kilocycle of sliding.



(b) 5 Kilocycles of sliding.

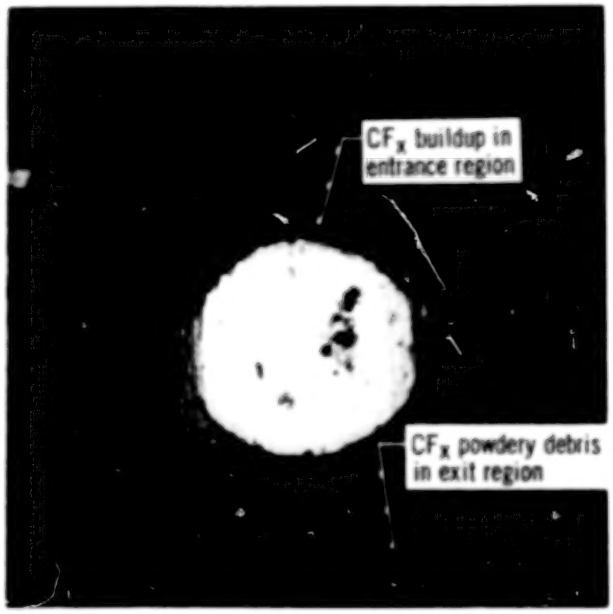


(c) 100 Kilocycles of sliding.

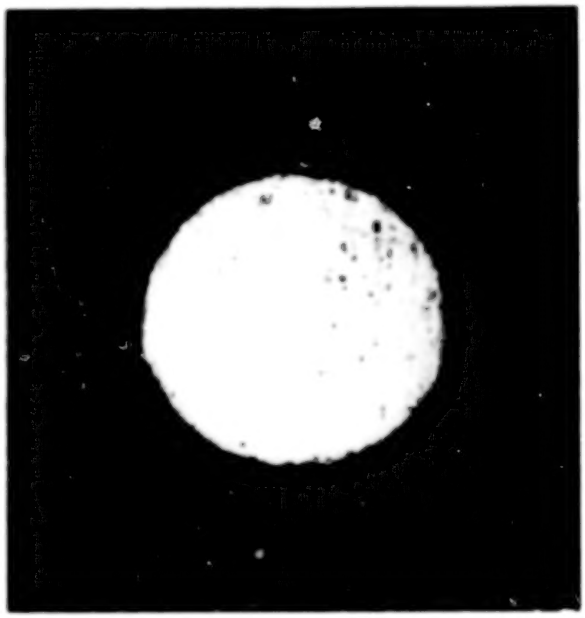


(d) 1200 Kilocycles of sliding.

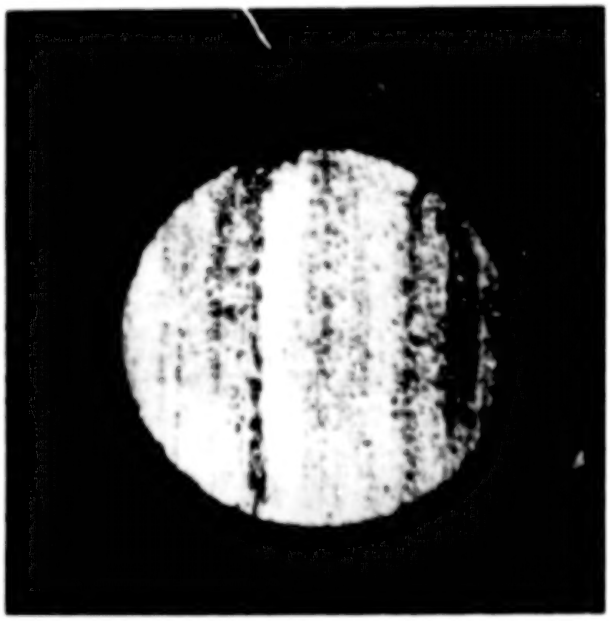
Figure 8. - Photomicrographs of central portion of film wear tracks for film applied to sandblasted 440C steel. Experimental conditions: load, 1 kilogram; sliding speed, 2.6 meters per second; test temperature, 25° C; test atmosphere, moist air (10 000 ppm H_2O).



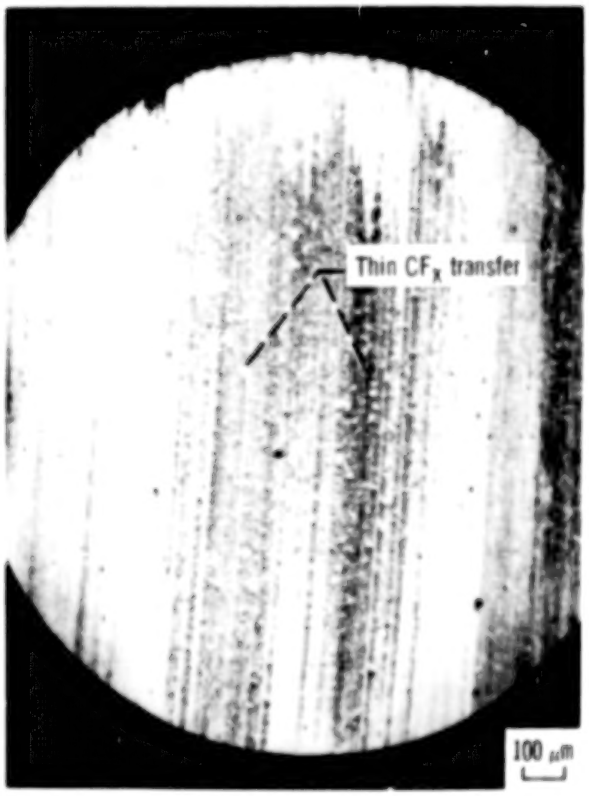
(a) 1 Kilocycle of sliding.



(b) 5 Kilocycles of sliding.

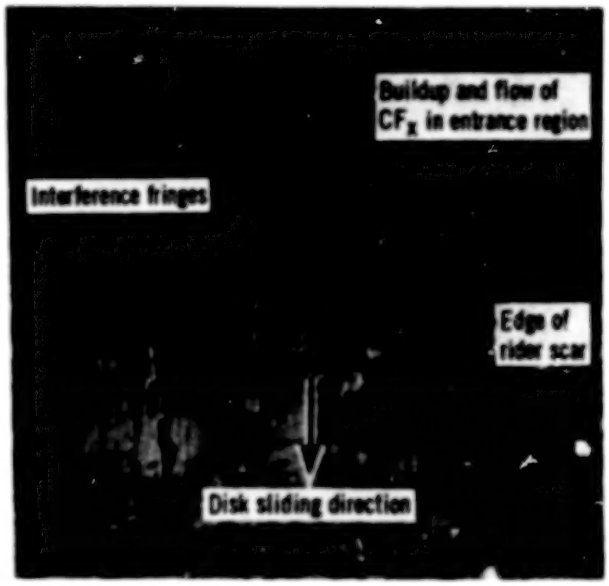


(c) 60 Kilocycles of sliding.

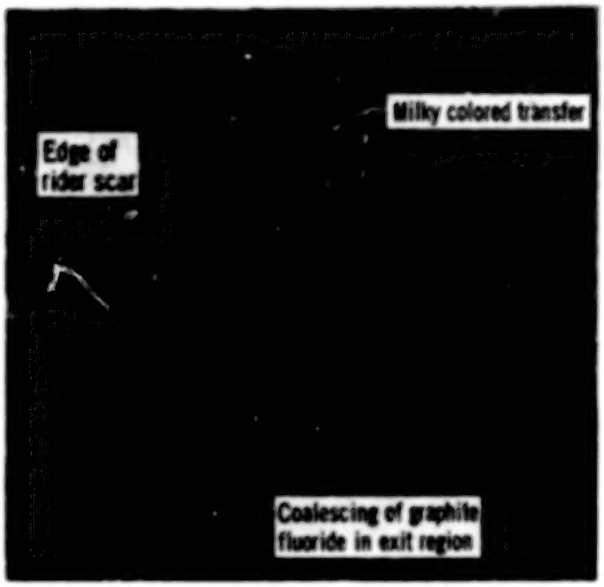


(d) 800 Kilocycles of sliding.

Figure 9. - Photomicrographs of 440C HT steel rider wear scars taken after various intervals of sliding on graphite fluoride films (applied to sandblasted 440C HT steel disks). Experimental conditions: load, 1 kilogram; sliding speed, 2.6 meters per second (1000 rpm); test temperature, 25° C; test atmosphere, moist air (10 000 ppm H_2O).



(a) Entrance area (1 kilocycle).

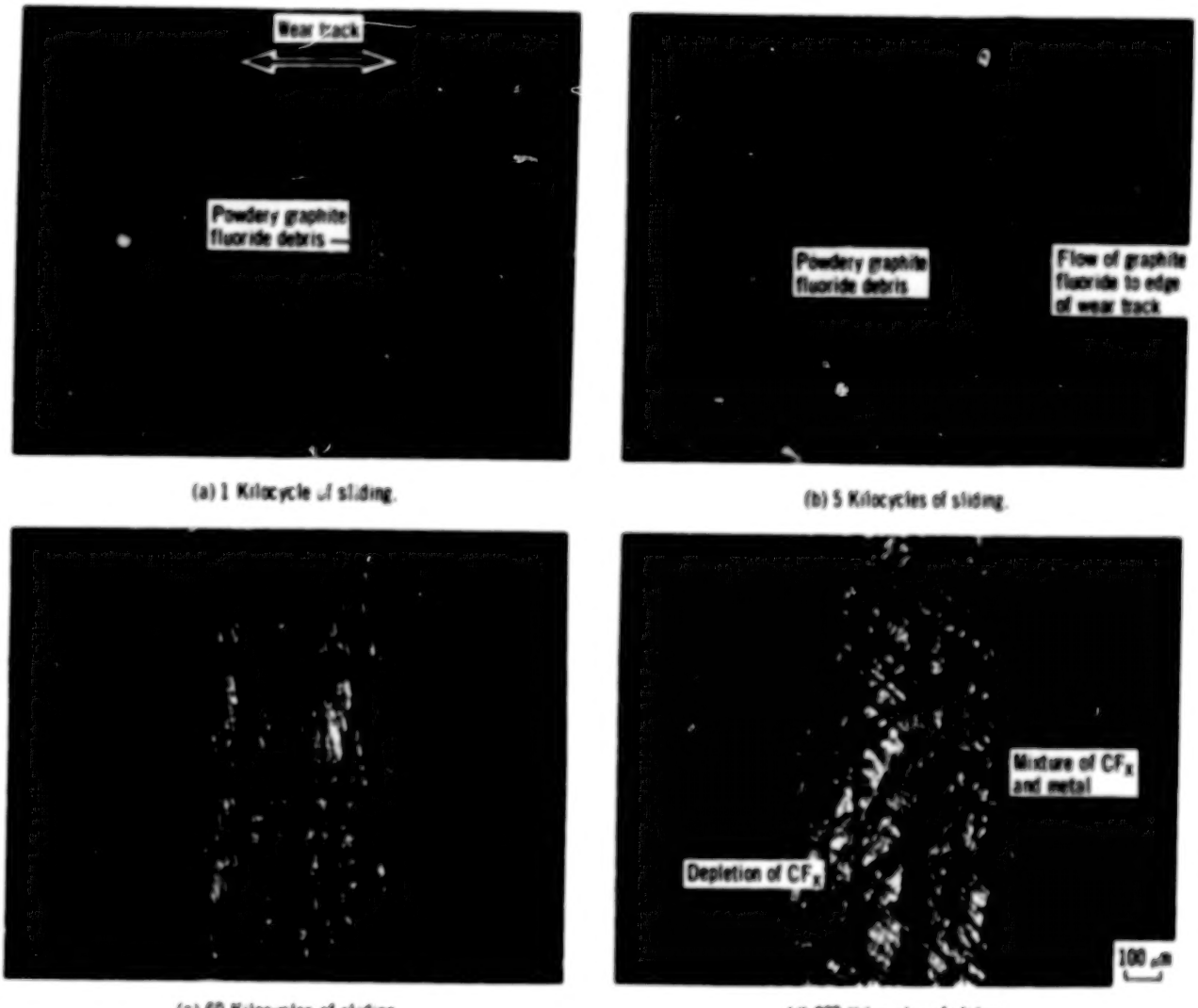


(b) Exit area (15 kilocycles).

(c) Middle of scar (60 kilocycles).

(d) Middle of scar (800 kilocycles).

Figure 10. - High magnification photomicrographs of typical transfer to various regions of the rider scar for riders which slid on graphite fluoride films applied to sandblasted disks. Experimental conditions: load, 1 kilogram; sliding speed, 2.6 meters per second (1000 rpm); test temperature, 25° C; test atmosphere, moist air (10 000 ppm H_2O).


Wear track
Powdery graphite fluoride debris
Powdery graphite fluoride debris
Flow of graphite fluoride to edge of wear track
Mixture of CF_x and metal
Depletion of CF_x
100 μm

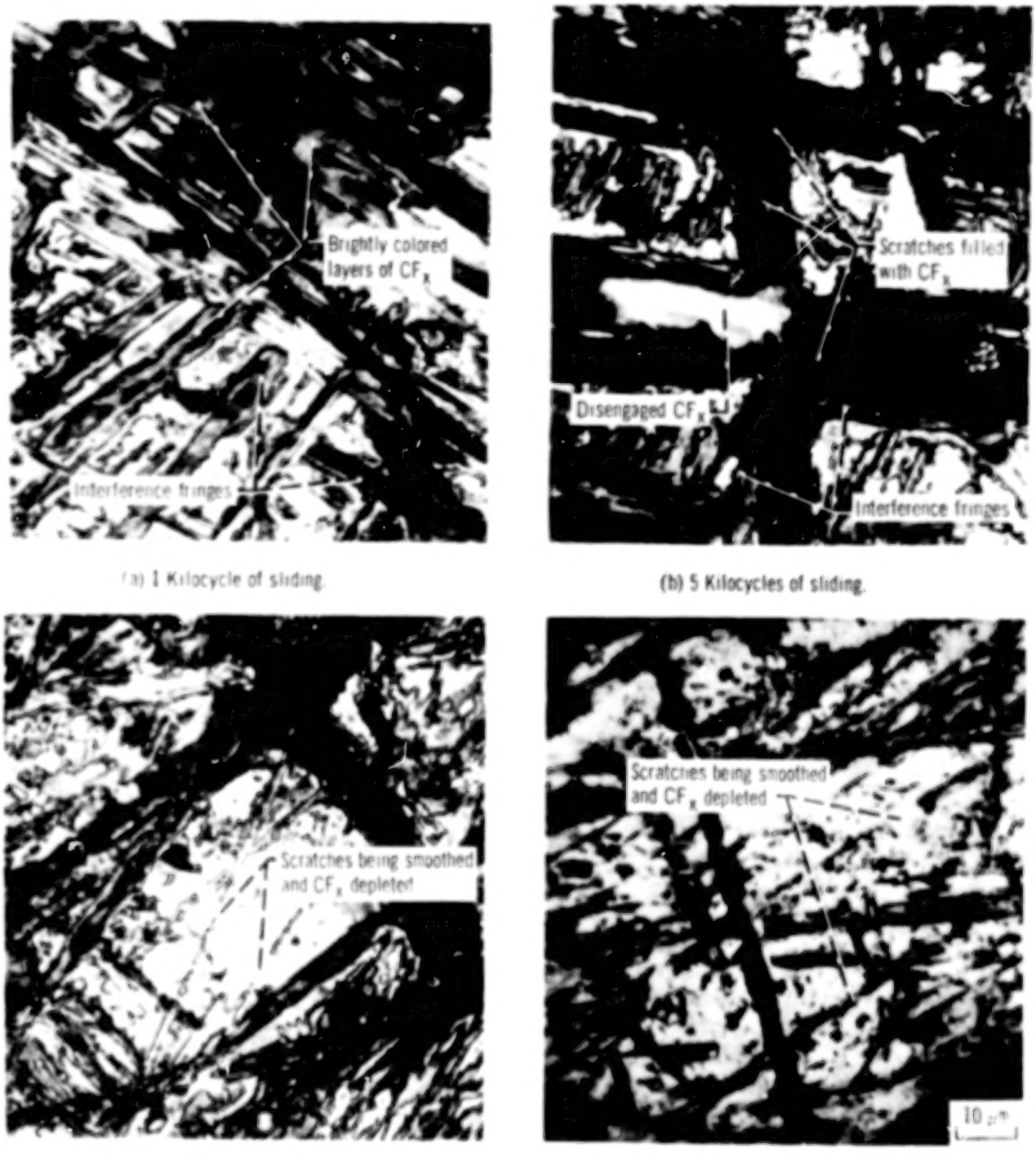
(a) 1 Kilocycle of sliding.

(b) 5 Kilocycles of sliding.

(c) 60 Kilocycles of sliding.

(d) 200 Kilocycles of sliding.

Figure 11. - Photomicrographs of wear tracks on graphite fluoride films applied to sanded 440C steel disks taken after various intervals of sliding. Experimental conditions: load, 1 kilogram; sliding speed, 2.6 meters per second; test temperature, 25° C; test atmosphere, moist air (10 000 ppm H_2O).



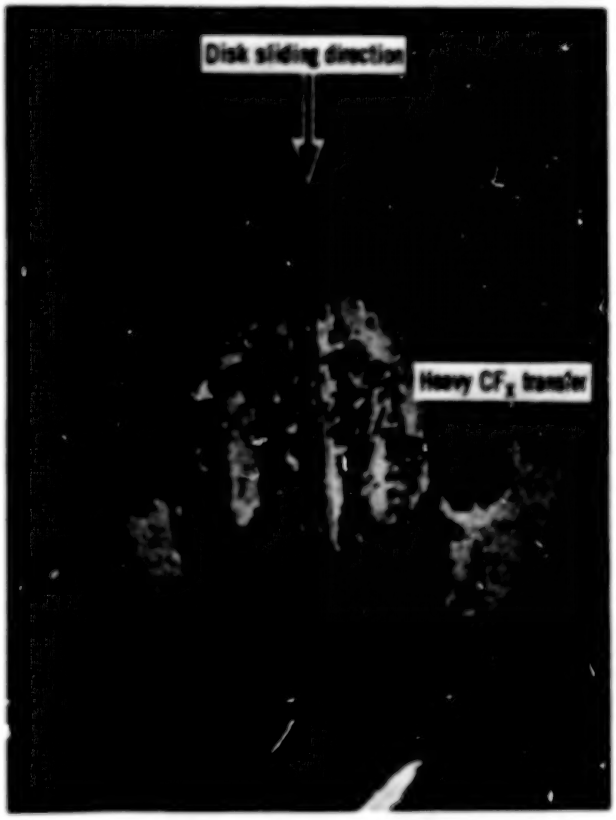
(a) 1 Kilocycle of sliding.

(b) 5 Kilocycles of sliding.

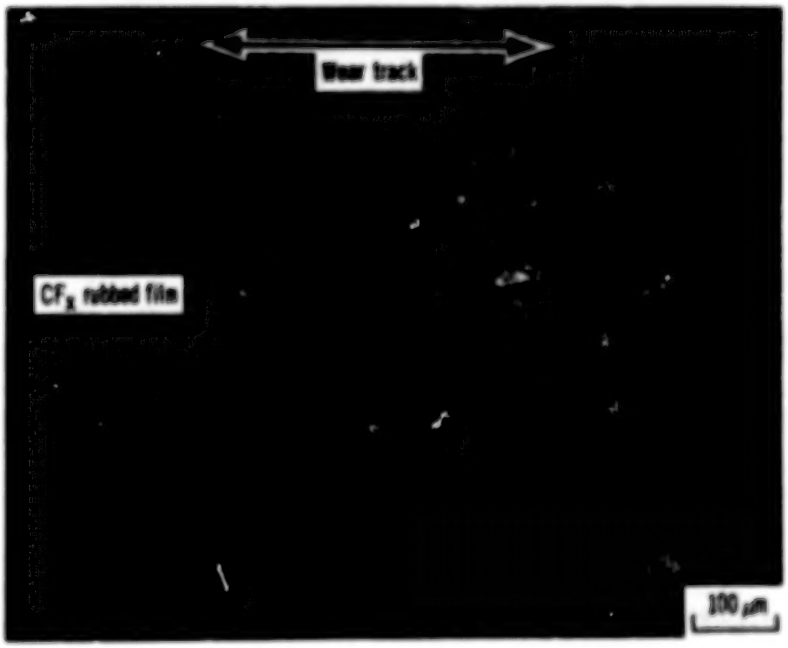
(c) 60 Kilocycles of sliding.

(d) 200 Kilocycles of sliding.

Figure 12. - High magnification photomicrographs of graphite fluoride film wear tracks on sanded 440C steel taken after various sliding intervals. Experimental conditions: load, 1 kilogram; sliding speed, 2.6 meters per second; test temperature, 25° C; test atmosphere, moist air (10 000 ppm H_2O).

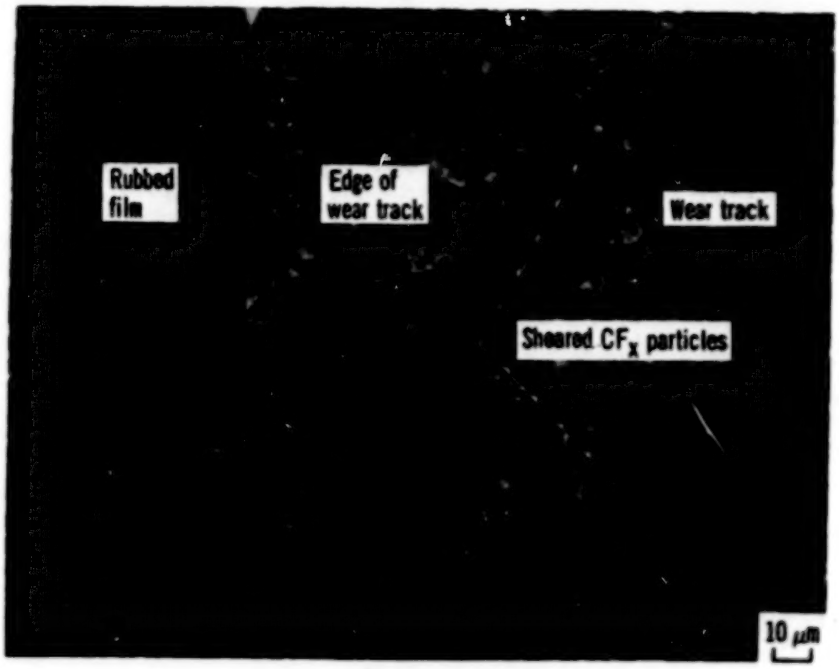


(a) Rider wear and transfer.

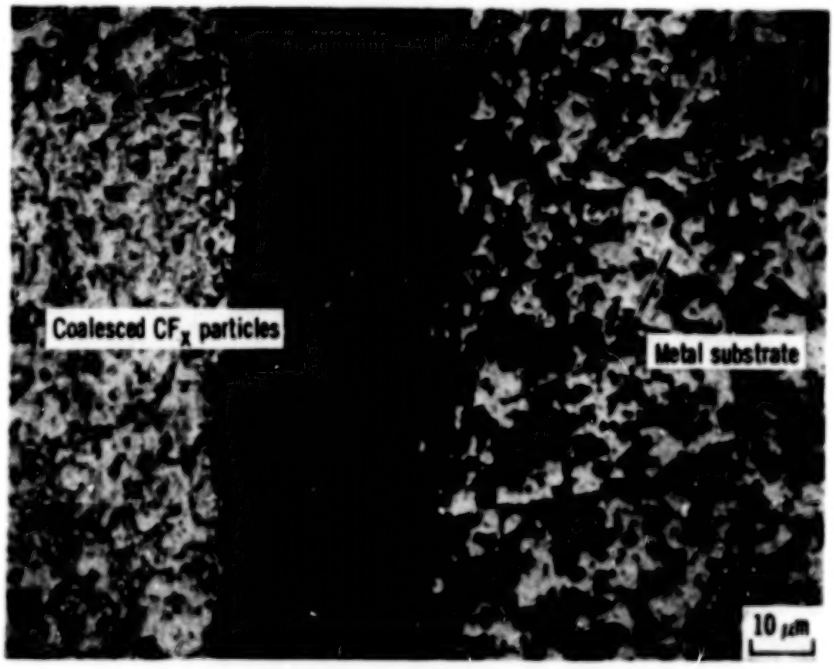


(b) Film wear track.

Figure 13. - Photomicrographs of rider wear and transfer and graphite fluoride wear tracks taken after 1 kilocycle of sliding on smooth 440C steel disks. Experimental conditions: load, 1 kilogram; sliding speed, 2.6 meters per second; test temperature 25° C; test atmosphere, moist air (10 000 ppm H_2O).

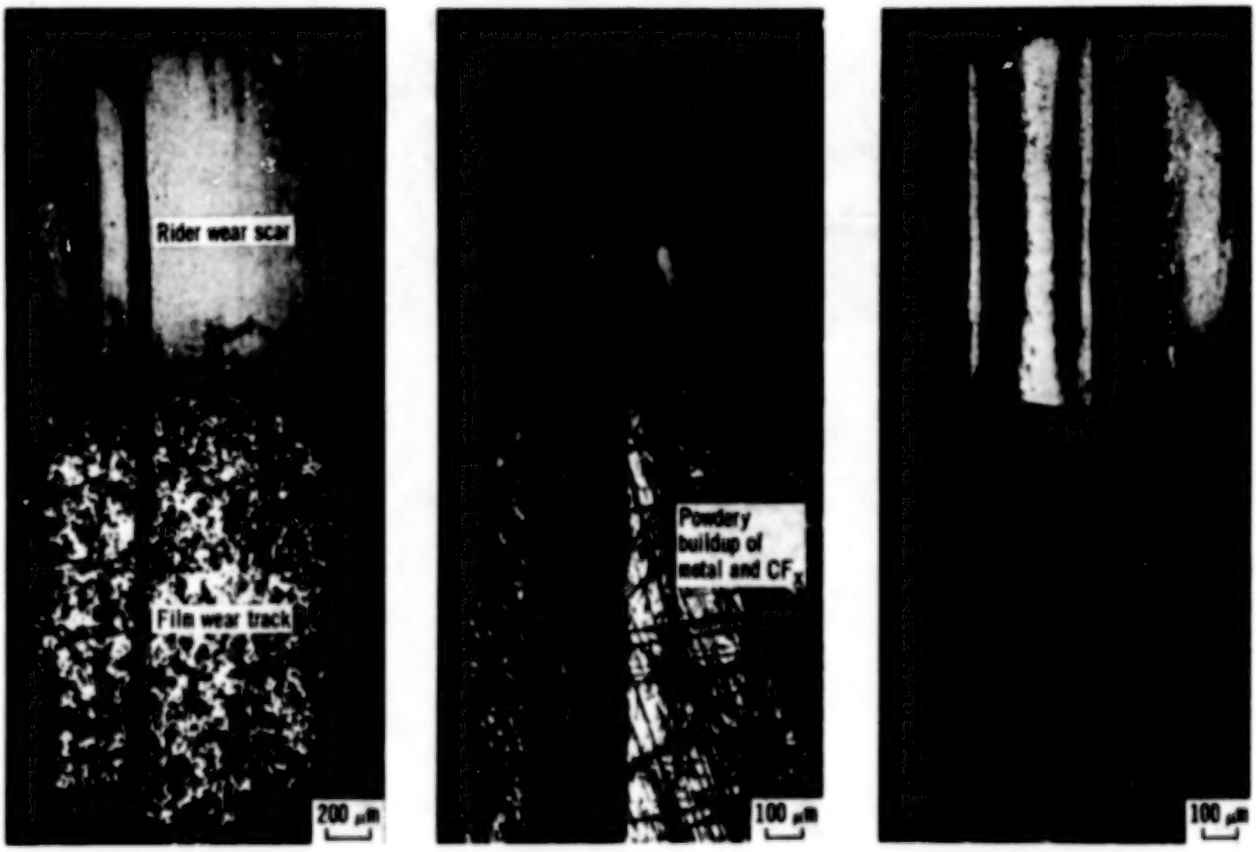


(a) 1 Kilocycle of sliding.



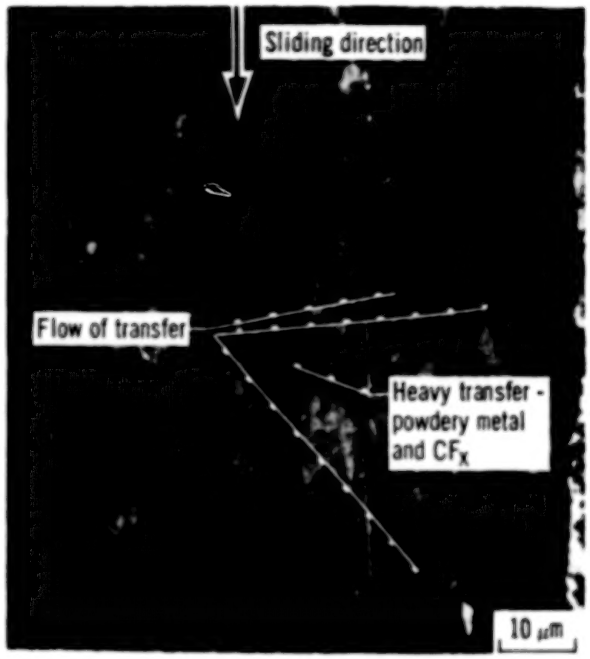
(b) 5 Kilocycles of sliding.

Figure 14. - High magnification photomicrographs of graphite fluoride film wear tracks on smooth 440C steel taken after various sliding intervals. Experimental conditions: load, 1 kilogram; sliding speed, 2.6 meters per second; test temperature, 25° C; test atmosphere, moist air (10 000 ppm H_2O).



(a) Sandblasted (1250 kilocycles). (b) Sanded (380 kilocycles). (c) Polished (42 kilocycles).

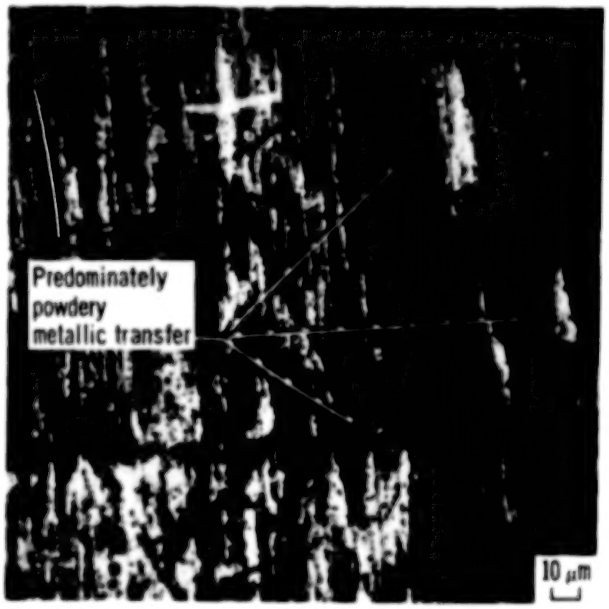
Figure 15. - Photomicrographs of rider wear scars and film wear tracks taken after failure and high friction have occurred. Experimental conditions: load, 1 kilogram; sliding speed, 2.6 meters per second (1000 rpm); test temperature, 25° C; test atmosphere, moist air (10 000 ppm H_2O).



(a) Rider transfer (300 kilocycles of sliding).

(b) Disk wear track (300 kilocycles of sliding).

(c) Rider transfer (380 kilocycles of sliding).



(d) Disk wear track (380 kilocycles of sliding).

Figure 16. - Typical transfer found of rider and disk (sanded) surfaces when high friction occurred. Experimental conditions: load, 1 kilogram; sliding speed, 2.6 meters per second (1000 rpm); test temperature, 25° C; atmosphere, moist air (10 000 ppm H_2O.

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16. Abstract

An optical microscope, equipped with a vertical illuminator and two polaroid filters (one rotatable), was used to visually study 440C HT steel surfaces lubricated with rubbed graphite fluoride films. Friction and wear results were compared to visual observations as a function of sliding distance for films applied to three surface finishes - polished, sanded, and sand-blasted. In general, the lubricating process was one of initial deformation or wear of metallic asperities into flat plateaus and then the formation of thin, layer-like, dynamic films which sheared between the flats and eventually flowed through the contact area. Failure was due to depletion of the graphite fluoride with the subsequent formation of excessive powdery metallic debris that formed a heavy, powdery film on both the rider and disk surfaces.

17. Key Words (Suggested by Author(s))	18. Distribution Statement
Graphite fluoride; $(CF_x)_n$; Solid lubricant; Lubrication mechanisms; Friction and wear surfaces; Surface pretreatments	Unclassified - unlimited STAR Category 27

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